EO US
PCT DK99 00286

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

Assistant Commissioner for Patents
United States Patent and Trademark Office
Box PCT
Washington, D.C. 20231
ETATS UNIS D'AMERIQUE

	02 December 1999 (02.12.99)
International application No. PCT/DK99/00286	International filing date (day/month/year) 21299 PC 1
International publication date (day/month/year) 27 May 1999 (27.05.99)	Priority date 27 May 1998 (27.05.98)
Applicant LUNDEN, Klaus, Akilles et al	

1. The designated Office is hereby notified of its election made

[X] in the demand filed with the International Preliminary Examining Authority on:

21 August 1999 (21.08.99)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] is
[ ] is not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Authorized officer

Copy for the Elected Office (EO/US)

PCT DK99 00286

PATENT COOPERATION TREATY

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From the INTERNATIONAL BUREAU

To:

PLOUGMANN, VINGTOFT & PARTNERS A S
Sankt Annæ Plads 11
P.O. Box 3007
DK-1021 Copenhagen K
DANEMARK

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

Date of mailing (day month year) 23 October 2000 (23.10.00)	
Applicant's or agent's file reference 21299 PC 1	**IMPORTANT NOTIFICATION**
International application No. PCT/DK99/00286	**International filing date** (day month year) 27 May 1999 (27.05.99)

1. The following indications appeared on record concerning:

[X] the applicant [ ] the inventor [ ] the agent [ ] the common representative

Name and Address	State of Nationality	State of Residence
FAXE PAPER PIGMENTS (DENMARK) A/S Strandesplanaden 110 DK-2665 Vallensbæk Strand Denmark	DK	DK
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

[ ] the person [X] the name [ ] the address [ ] the nationality [ ] the residence

Name and Address	State of Nationality	State of Residence
J.M. HUBER DENMARK APS Strandesplanaden 110 DK-2665 Vallensbæk Strand Denmark	DK	DK
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

[X] the receiving Office
[ ] the International Searching Authority
[ ] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Authorized officer:
S. De Michelis

Facsimile No. (41-22) 740 14 35

Telephone No. (41-22) 338 83 38

Form PCT/IB/306 (March 1994)

PATENT COOPERATION TREATY

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	**FOR FURTHER ACTION**	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
21299 PC 1		
International application No.	International filing date *(day/month/year)*	(Earliest) Priority Date *(day/month/year)*
PCT/DK 99/ 00286	27/05/1999	27/05/1998
Applicant		
FAXE PAPER PIGMENTS (DENMARK) A/S et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1 **Basis of the report**

a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

[ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

[ ] contained in the international application in written form.

[ ] filed together with the international application in computer readable form.

[ ] furnished subsequently to this Authority in written form.

[ ] furnished subsequently to this Authority in computer readble form.

[ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

[ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).

3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,

[ ] the text is approved as submitted by the applicant.

[X] the text has been established by this Authority to read as follows:

USE OF COLLOIDAL PRECIPITATED CALCIUM CARBONATE AS A FILTER IN THE PREPARATION OF PAPER

5. With regard to the **abstract**,

[X] the text is approved as submitted by the applicant.

[ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

[ ] as suggested by the applicant.

[ ] because the applicant failed to suggest a figure.

[ ] because this figure better characterizes the invention.

[X] None of the figures.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
21299 PC 1		
International application No	International filing date (day month year)	Priority date (day month year)
PCT/DK99/00286	27/05/1999	27/05/1998

International Patent Classification (IPC) or national classification and IPC
D21H23/12

Applicant
FAXE PAPER PIGMENTS (DENMARK) A/S et al.

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

   [X] This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 5 sheets.

3. This report contains indications relating to the following items:

   I [X] Basis of the report
   II [ ] Priority
   III [ ] Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   IV [ ] Lack of unity of invention
   V [X] Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   VI [X] Certain documents cited
   VII [X] Certain defects in the international application
   VIII [X] Certain observations on the international application

Date of submission of the demand	Date of completion of this report
21/08/1999	22.08.00

Name and mailing address of the international preliminary examining authority:
European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer
Naeslund, P

Telephone No. +49 89 2399 8614

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/DK99/00286

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1,2,4-17	as originally filed			
3,3a	as received on	08/01/2000	with letter of	05/01/2000

Claims, No.:

1-23	as received on	08/01/2000	with letter of	05/01/2000

Drawings, sheets:

1/2,2/2	as originally filed

2. The amendments have resulted in the cancellation of:

☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-23
	No:	Claims	None
Inventive step (IS)	Yes:	Claims	None
	No:	Claims	1-23
Industrial applicability (IA)	Yes:	Claims	1-23
	No:	Claims	None

2. Citations and explanations

see separate sheet

VI. Certain documents cited

1. Certain published documents (Rule 70.10)

and / or

2. Non-written disclosures (Rule 70.9)

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Re Item V

Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. From **US-A-4 892 590 (D1)**(see col. 3, line 24-line 29; col. 3, line 57-col. 4, line 24) cited by the applicant in the description of the present application, there is (implicitly) known a process for regulating the porosity and printing properties of uncoated paper, the process comprising using a sufficient quantity of colloidal PCC having a BET surface area of 10-100m^2/g as a filler to achieve a desired porosity of the paper. Hence, the only feature of claim 1 which is **not** known from **D1** is that the paper is "wood containing". The furnish used in **D1** is based on chemical pulps. No inventive step, however, can be accorded claim 1 because of this difference, as wood containing (mechanical) and chemical pulps are interchangeably used by the skilled person involved in the production of printing paper. Moreover, no particular effects can be seen related to the stock composition. Claim 1 therefore would not appear inventive; Art. 33(3) PCT.
Since the method of manufacturing the paper is not inventive, the products obtained therewith are not inventive either. Thus, product claims 9, 14, 16 and 17 would not appear inventive either (Art. 33(3) PCT).

2. In view of **WO-A-9 629 369 (D2)** (see claims 14 and 15; example 6), pigment mixtures as claimed in claims 20 - 23 would not appear inventive either (Art. 33(3) PCT).

3. By the same token, in view of the cited prior art, **WO-A-97 30220 (D3)** (see page 5, paragraph 4-page 6, paragraph 1; page 8, line 25-page 9, line 8; claims 1, 2 and 4) and **EP-A-0 521 737(D4)** (see page 2, line 5; page 2, line 34-line 37) inclusively, the rest of the dependent claims would only appear to relate to known and/or trivial matter (Art. 33(3) PCT).

4. The industrial applicability would appear evident (Art. 33(4) PCT).

Re Item VI

Certain documents cited

EP-A-0 850 880 (see page 5, line 47-line 53; claim 6) is filed on 23.12.1997, claims a priority from 27.12.1996 and was published on 01.07.1998. It appears to disclose the subject-matters of at least claims 10-13 and 22. This application which claims an earlier priority date than the present application constitutes a potential prior right on entry of the European phase. No check has been made as to whether the priority of the present application has been validly claimed. The priority document is not yet available.

Re Item VII

Certain defects in the international application

1. Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents **D2-D4** are not mentioned in the description, nor are these documents identified therein.

2. In case that the two-part form is not applied, the applicant should provide reasons why it would not appear appropriate in the present case. In any case he should clearly indicate in the description which features of the subject-matter of in particular the independent claims are already known from the closest prior art.

Re Item VIII

Certain observations on the international application

1. Claims should be clear taken alone. It is not clear what is meant by SC,SC-B in inter alia claims 2,3,14,15 and 16 (Art. 6 PCT).

2 Further, parameters e.g. opacity should be avoided in product claims. At the entry of the national/regional phase, it might be necessary to file comparative tests, in case novelty is relied upon solely on parameters such as indicated.

0.20 µm, e.g. colloidal particles of at most 0.01 µm; such colloidal particles can be colloidal calcium carbonate.

WO 96/29369 discloses an ink jet recording paper with at least one side coated with a coating composition comprising heat aged and/or milled PCC and a binder.

WO 97/30220 discloses production of filled papers using a cellulosic suspension comprising a slurry of PCC and a cationic polymer.

EP 0 521 737-A1 discloses a method for increasing the solids content of a PCC slurry for use in paper manufacture by adding to the slurry a powdered or granulated pigment.

It is not believed that colloidal PCC has previously been described or used as a filler in paper for the purpose of controlling the porosity and printing properties of the paper.

DESCRIPTION OF THE INVENTION

It has now been found that use of colloidal PCC with a large surface area as a filler makes it possible to replace a proportion of the previously mentioned pigments whilst also providing the possibility of regulating the porosity and printability properties of the paper. Compared with the previously described methods, the use of colloidal PCC has numerous advantages. It is cheap, produces low wear, it can produce greater brightness than kaolin and talc flakes, and the product is more adaptable to individual types of paper.

In its broadest aspect, the present invention relates to the use of colloidal PCC as a filler to control the porosity and printing properties of paper, in particular to reduce the porosity relative to the porosity which can otherwise be achieved with other types of fillers and pigments conventionally used in the manufacture of paper.

One aspect of the invention thus relates to a process for regulating the porosity and printing properties of paper, wherein a sufficient quantity of colloidal PCC having a

BET surface area of 10-100 m^2/g is used as a filler to achieve a desired porosity of the paper.

In another aspect, the invention relates to paper containing colloidal PCC as a filler.

In a third aspect, the invention relates to a pigment mixture which is suitable for manufacture of paper and which contains colloidal PCC.

Other aspects and preferred embodiments will be apparent from the following detailed description of the invention.

Amended claims

1. A process for regulating the porosity and printing properties of uncoated wood-containing paper, the process comprising using a sufficient quantity of colloidal PCC having a BET surface area of 10-100 m^2/g as a filler to achieve a desired porosity of the paper.

2. A process according to claim 1, wherein the paper is SC paper, in particular SC-A paper, and wherein colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.30 µm/Pas, e.g. at most 0.28 µm/Pas, e.g. at most 0.26 µm/Pas, e.g. at most 0.24 µm/Pas, e.g. at most 0.22 µm/Pas.

3. A process according to claim 1, wherein the paper is SC-B paper, and wherein colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.60 µm/Pas, e.g. at most 0.50 µm/Pas, e.g. at most 0.40 µm/Pas, e.g. at most 0.35µm/Pas.

4. A process according to claim 1, wherein the paper is newsprint, and wherein colloidal PCC is used in an amount sufficient to achieve a porosity of at most 20 µm/Pas, e.g. at most 18 µm/Pas, e.g. at most 16 µm/Pas.

5. A process according to any of the preceding claims, wherein the colloidal PCC has a BET surface area of 15-50 m^2/g.

6. A process according to claim 5, wherein the colloidal PCC has a BET surface area of 20-30 m^2/g.

7. A process according to any of the preceding claims, wherein colloidal PCC is incorporated into the paper in an amount of at least about 1% by weight based on the total weight of the paper.

8. A process according to claim 7, wherein colloidal PCC is incorporated into the paper in an amount of at least about 2% by weight based on the total weight of the paper.

9. Uncoated wood-containing paper containing colloidal PCC.

10. Paper according to claim 9 containing colloidal PCC having a BET surface area of 10-100 m^2/g as a filler.

11. Paper according to claim 10, comprising at least one further filler selected from non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

12. Paper according to claim 10 or 11, wherein the colloidal PCC has a BET surface area of 15-50 m^2/g, e.g. 20-30 m^2/g.

13. Paper according to any of claims 9-12, wherein the colloidal PCC is present in an amount of at least about 1% by weight, e.g. at least about 2% by weight, based on the total weight of the paper.

14. SC paper containing colloidal PCC and having a porosity of at most 0.30 µm/Pas, e.g. at most 0.28 µm/Pas, e.g. at most 0.26 µm/Pas, e.g. at most 0.24 µm/Pas, e.g. at most 0.22 µm/Pas.

15. SC paper according to claim 14, wherein the paper is SC-A paper.

16. SC-B paper containing colloidal PCC and having a porosity of at most 0.60 µm/Pas, e.g. at most 0.50 µm/Pas, e.g. at most 0.40 µm/Pas, e.g. at most 0.35 µm/Pas.

17. Newsprint containing colloidal PCC and having a porosity of at most 20 µm/Pas, e.g. at most 18 µm/Pas, e.g. at most 16 µm/Pas.

18. Paper according to any of claims 14-17, comprising at least one further filler selected from non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

19. Paper according to any of claims 14-18, wherein the colloidal PCC has a BET surface area of 10-100 m^2/g, e.g. 15-50 m^2/g, e.g. 20-30 m^2/g.

20. A pigment mixture suitable for paper manufacture and comprising colloidal PCC having a BET surface area of 10-100 m^2/g in combination with at least one filler selected from the following pigments: kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

21. A pigment mixture suitable for paper manufacture and comprising a combination of colloidal PCC having a BET surface area of 10-100 m^2/g and non-colloidal PCC.

22. A pigment mixture according to claim 20 or 21, wherein the colloidal PCC has a BET surface area of 15-50 m^2/g, e.g. 20-30 m^2/g.

23. A pigment mixture according to any of claims 20-22, wherein the colloidal PCC comprises aggregates/agglomerates having an equivalent spherical particle size in the range 0.1-5.0 µm, e.g. 0.2-4 µm, typically 0.5-3.0 µm, wherein the aggregates/agglomerates consist of single crystals having an equivalent spherical particle size of about 0.01-0.50 µm.

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference *(if desired) (12 characters maximum)* 21299 PC 1

Box No. I TITLE OF INVENTION

USE OF COLLOIDAL PCC

Box No. II APPLICANT

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Faxe Paper Pigments (Denmark) A/S
Strandesplanaden 110
DK-2665 Vallensbæk Strand
Denmark

☐ This person is also inventor

Telephone No.

Facsimile No.

Teleprinter No.

State *(that is, country)* of nationality: Denmark

State *(that is, country)* of residence: Denmark

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Lundén, Klaus Akilles
Byengen 50
DK-2980 Kokkedal
Denmark

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only *(If this check-box is marked, do not fill in below)*

State *(that is, country)* of nationality: Denmark

State *(that is, country)* of residence: Denmark

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☒ Further applicants and or (further) inventors are indicated on a continuation sheet

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

Plougmann, Vingtoft & Partners A/S
Sankt Annæ Plads 11
P.O. Box 3007
DK-1021 Copenhagen K
Denmark

Telephone No.
+45 33 63 93 00

Facsimile No.
+45 33 63 96 00

Teleprinter No.

☐ **Address for correspondence:** Mark this check-box where no agent or common representative is has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Attrup, Ib
Grønlykkeparken 11F
DK-2670 Greve
Denmark

This person is:
- ☐ applicant only
- ☒ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: Denmark

State *(that is, country)* of residence: Denmark

This person is applicant for the purposes of:
- ☐ all designated States
- ☐ all designated States except the United States of America
- ☒ the United States of America only
- ☐ the States indicated in the Supplemental Box

Name and address *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Madsen, Jens Toftelund
Plantevej 15, 4 th
DK-2860 Søborg
Denmark

This person is:
- ☐ applicant only
- ☒ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: Denmark

State *(that is, country)* of residence: Denmark

This person is applicant for the purposes of:
- ☐ all designated States
- ☐ all designated States except the United States of America
- ☒ the United States of America only
- ☐ the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of:
- ☐ all designated States
- ☐ all designated States except the United States of America
- ☐ the United States of America only
- ☐ the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of:
- ☐ all designated States
- ☐ all designated States except the United States of America
- ☐ the United States of America only
- ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on another continuation sheet

Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) *(mark the applicable check-boxes; at least one must be marked)*:

Regional Patent

- [X] AP **ARIPO Patent:** GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- [X] EA **Eurasian Patent:** AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- [ ] EP **European Patent:** AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- [ ] OA **OAPI Patent:** BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT *(if other kind of protection or treatment desired, specify on dotted line)* .....

National Patent *(if other kind of protection or treatment desired, specify on dotted line)*

- [ ] AL Albania .....
- [ ] AM Armenia .....
- [X] AT Austria ...and utility model...
- [X] AU Australia .....
- [X] AZ Azerbaijan
- [X] BA Bosnia and Herzegovina .....
- [X] BB Barbados
- [X] BG Bulgaria .....
- [X] BR Brazil .....
- [X] BY Belarus .....
- [X] CA Canada
- [X] CH and LI Switzerland and Liechtenstein
- [X] CN China .....
- [X] CU Cuba .....
- [X] CZ Czech Republic and utility model
- [X] DE Germany ...and utility model...
- [X] DK Denmark ...and utility model...
- [X] EE Estonia ...and utility model...
- [X] ES Spain .....
- [X] FI Finland ...and utility model...
- [X] GB United Kingdom
- [X] GD Grenada
- [X] GE Georgia .....
- [X] GH Ghana .....
- [X] GM Gambia
- [X] HR Croatia .....
- [X] HU Hungary .....
- [X] ID Indonesia
- [X] IL Israel .....
- [X] IN India .....
- [X] IS Iceland
- [X] JP Japan .....
- [X] KE Kenya .....
- [X] KG Kyrgyzstan .....
- [X] KP Democratic People's Republic of Korea .....
- [X] KR Republic of Korea .....
- [X] KZ Kazakhstan .....
- [X] LC Saint Lucia
- [X] LK Sri Lanka
- [X] LR Liberia
- [X] LS Lesotho .....
- [X] LT Lithuania
- [X] LU Luxembourg
- [X] LV Latvia
- [X] MD Republic of Moldova .....
- [X] MG Madagascar .....
- [X] MK The former Yugoslav Republic of Macedonia .....
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See Notes to the request form

Box No. VI PRIORITY CLAIM ☐ Further priority claims are indicated in the Supplemental Box.

Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is: national application: country	regional application:* regional Office	international application: receiving Office
item (1) 27 May 1998	PA 1998 00735	Denmark		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *(only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office)* identified above as item(s) (1)

* *Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(iii)). See Supplemental Box*

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) *(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used)*: ISA / EP

Request to use results of earlier search; reference to that search *(if an earlier search has been carried out by or requested from the International Searching Authority)*: Date *(day/month/year)* Number Country *(or regional Office)*

Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following **number of sheets**:

request	4
description (excluding sequence listing part)	17
claims	3
abstract	1
drawings	2
sequence listing part of description	
Total number of sheets	27

This international application is **accompanied by** the item(s) marked below:

1. ☒ fee calculation sheet
2. ☐ separate signed power of attorney
3. ☐ copy of general power of attorney; reference number, if any:
4. ☐ statement explaining lack of signature
5. ☐ priority document(s) identified in Box No. VI as item(s):
6. ☐ translation of international application into (language):
7. ☐ separate indications concerning deposited microorganism or other biological material
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9. ☐ other *(specify)*:

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: English

Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request)

Copenhagen, 27 May 1999
Plougmann, Vingtoft & Partners A/S

Jeff Salka

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3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:
4. Date of timely receipt of the required corrections under PCT Article 11(2):
5. International Searching Authority (if two or more are competent): ISA /
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2. Drawings:
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☐ not received:

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/DK 99/00286

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 D21H23/12 D21H17/67 D21H17/71 D21H21/22 D21H27/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 892 590 A (GILL ROBERT A ET AL) 9 January 1990 cited in the application see column 3, line 24 - line 29 see column 3, line 57 - column 4, line 24	1,6,7, 10-13
A	---	2-5,8,9, 14-25
A	US 4 460 637 A (MIYAMOTO SHIGEHIKO ET AL) 17 July 1984 cited in the application see the whole document	1-25
P,X	---	10-12, 14,22
	EP 0 850 880 A (OKUTAMA KOGYO CO LTD) 1 July 1998 see page 5, line 47 - line 53 see claim 6	

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[X] Further documents are listed in the continuation of box C

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15 July 1999	22/07/1999

Name and mailing address of the ISA	Authorized officer
European Patent Office, P B 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016	Naeslund, P

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/DK 99/00286

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
X	WO 96 29369 A (MINERALS TECH INC) 26 September 1996 see claims 14,15 see example 6	10,12,14
A		1-9,11, 13,15-25

A	WO 97 30220 A (ALLIED COLLOIDS LTD ;MINERALS TECH INC (US); DEPASQUALE DAVID (CA)) 21 August 1997 see page 5, paragraph 4 - page 6, paragraph 1 see page 8, line 25 - page 9, line 8 see claims 1,2,4	3-5, 8-11, 14-16, 18-20, 22,23

A	EP 0 521 737 A (PARTEK AB) 7 January 1993 see page 2, line 5 see page 2, line 34 - line 37	1,8,9, 11,22

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/DK 99/00286

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PCT/DK 99/00286

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Minimum documentation searched (classification system followed by classification symbols)
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A		2-5,8,9, 14-25

A	US 4 460 637 A (MIYAMOTO SHIGEHIKO ET AL) 17 July 1984 cited in the application see the whole document	1-25

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Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/DK 99/00286

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		DK 272089 A	04-12-1989
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EP 0521737 A	07-01-1993	FI 913255 A	05-01-1993

CORRECTED VERSION*

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 :
D21H 23/12, 17/67, 17/71, 21/22, 27/00

A1

(11) International Publication Number: WO 99/61703

(43) International Publication Date: 2 December 1999 (02.12.99)

(21) International Application Number: PCT/DK99/00286

(22) International Filing Date: 27 May 1999 (27.05.99)

(30) Priority Data:
PA 1998 00735 27 May 1998 (27.05.98) DK

(71) Applicant *(for all designated States except US)*: FAXE PAPER PIGMENTS (DENMARK) A/S [DK/DK]; Strandesplanaden 110, DK–2665 Vallensbæk Strand (DK).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: LUNDÉN, Klaus, Akilles [DK/DK]; Byengen 50, DK–2980 Kokkedal (DK). ATTRUP, Ib [DK/DK]; Grønlykkeparken 11F, DK–2670 Greve (DK). MADSEN, Jens, Toftelund [DK/DK]; Plantevej 15, 4 th, DK–2860 Søborg (DK).

(74) Agent: PLOUGMANN, VINGTOFT & PARTNERS A/S; Sankt Annæ Plads 11, P.O. Box 3007, DK–1021 Copenhagen K (DK).

(81) Designated States: AE, AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), EE, EE (Utility model), ES, FI, FI (Utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: USE OF COLLOIDAL PRECIPITATED CALCIUM CARBONATE AS A FILLER IN THE PREPARATION OF PAPER

(57) Abstract

The invention relates to a process for regulating the porosity and printing properties of paper, in particular uncoated wood–containing paper such as SC–paper, wherein a sufficient quantity of colloidal PCC having a BET surface area of 10–100 m^2/g is used as a filler in the paper to achieve a desired porosity of the paper; as well as paper containing colloidal PCC as filler, and a pigment mixture suitable for paper manufacture and containing colloidal PCC.

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 :
D21H 23/12, 17/67, 17/71, 21/22, 27/00

A1

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(81) Designated States: AE, AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), EE, EE (Utility model), ES, FI, FI (Utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

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(57) Abstract

The invention relates to a process for regulating the porosity and printing properties of paper, in particular uncoated wood–containing paper such as SC–paper, wherein a sufficient quantity of colloidal PCC having a BET surface area of 10–100 m^2/g is used as a filler in the paper to achieve a desired porosity of the paper; as well as paper containing colloidal PCC as filler, and a pigment mixture suitable for paper manufacture and containing colloidal PCC.

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USE OF COLLOIDAL PRECIPITATED CALCIUM CARBONATE AS A FILTER IN THE PREPARATION OF PAPER

FIELD OF THE INVENTION

The invention relates to use of colloidal PCC (precipitated calcium carbonate) as a filler in the preparation of paper for the purpose of controlling the porosity and printing properties of the paper.

BACKGROUND OF THE INVENTION

In the connection with the manufacture of paper it is very important to be able to control the porosity of the paper. For example, a paper with low porosity is required in order to obtain an acceptable result in, e.g., ink-jet and rotogravure printing. If the paper is too porous it will function like blotting paper during printing and the resulting print may appear blurred, the contrast between printed and unprinted areas or between differently coloured areas not being rendered sharply. Similarly, on a paper which is of non-uniform porosity it can be seen that the intensity of colouration varies ("mottling"), which is of course undesirable since the coloured surface appears variegated or mottled. On the other hand, the porosity of the paper can also be too low, since a very dense paper will have difficulty in absorbing printing ink, which among other things may result in smudging ("set off") between printed sheets. This phenomenon can influence the printing results, the printing speed and the printing process employed in a negative manner.

The paper industry presently uses several different ways of regulating the porosity of paper. Use is made among other things of the fact that certain minerals in the form of flakes, e.g. talc and kaolin, will, as result of their form, be able to reduce the porosity since the individual particles will become deposited like the scales on a fish and thereby seal the surface. Fine silicates can be used in connection with pigmentation to reduce the porosity of the paper. When they come into or onto the paper, these fine particles will close the pores which contribute to the porosity of the paper.

In order to regulate the properties of the paper, a combination of one or more fillers and a variety of other additives is often used. Among the group of additives are

alkylketene dimers (AKD), alkenylsuccinic acid anhydride (ASA), starch and retention agents. Retention agents are added to facilitate the manufacture of the paper, whilst AKD, ASA and starch are added to ensure the quality of the paper (strength, printing properties, etc.).

Regardless of which of the presently known methods is used, they all have drawbacks. Kaolin and talc in the form of flakes will negatively influence the brightness of the paper compared to the whiter fillers, such as ground marble or PCC (precipitated calcium carbonate).

The fine silicate products used for pigmentation have relatively good technical properties. However, the silicate products have the disadvantage of being much more expensive than the fillers normally used in paper manufacture. The same applies to other additives normally used in connection with paper manufacture. These are often many times more expensive than a calcium carbonate filler.

Over the years, numerous attempts have been made to optimise paper compositions precisely for the purpose of improving the porosity and printing properties of the paper. The problem has been, however, that none of these approaches to a solution have been ideal, since they have either had a negative influence on the other properties of the paper (among other things the brightness) or are relatively expensive to use (silicate products).

The use of colloidal PCC as such in paper is known. For example, US 4,892,590 discloses the use of a two-component binder system as a retention agent for paper manufacture, wherein the binder comprises colloidal PCC with a high specific surface area together with a cationic starch. The PCC used has a surface area of 10-200 m^2/g, and the weight ratio between PCC and cationic starch is from 2:1 to 1:20.

US 4,460,637 discloses ink-jet paper (coated paper) with 2 different peaks of pore size distribution in the ink-receiving layer or layers. The desired pore size distribution can be achieved, inter alia by means of agglomerates with an average diameter of 1-50 µm in which the individual particles in the agglomerates have a size of at most

0.20 µm, e.g. colloidal particles of at most 0.01 µm; such colloidal particles can be colloidal calcium carbonate.

It is not believed that colloidal PCC has previously been described or used as a filler in paper for the purpose of controlling the porosity and printing properties of the paper.

DESCRIPTION OF THE INVENTION

It has now been found that use of colloidal PCC with a large surface area as a filler makes it possible to replace a proportion of the previously mentioned pigments whilst also providing the possibility of regulating the porosity and printability properties of the paper. Compared with the previously described methods, the use of colloidal PCC has numerous advantages. It is cheap, produces low wear, it can produce greater brightness than kaolin and talc flakes, and the product is more adaptable to individual types of paper.

In its broadest aspect, the present invention relates to the use of colloidal PCC as a filler to control the porosity and printing properties of paper, in particular to reduce the porosity relative to the porosity which can otherwise be achieved with other types of fillers and pigments conventionally used in the manufacture of paper.

One aspect of the invention thus relates to a process for regulating the porosity and printing properties of paper, wherein a sufficient quantity of colloidal PCC having a BET surface area of 10-100 m^2/g is used as a filler to achieve a desired porosity of the paper.

In another aspect, the invention relates to paper containing colloidal PCC as a filler.

In a third aspect, the invention relates to a pigment mixture which is suitable for manufacture of paper and which contains colloidal PCC.

Other aspects and preferred embodiments will be apparent from the following detailed description of the invention.

As employed in the present description and claims, the term "colloidal PCC" (chemical formula: $CaCO_3$) designates a PCC product in the form of aggregates/agglomerates of individual PCC particles in which the aggregates/agglomerates have a surface area of at least 10 m^2/g as determined by the BET method (Brunauer, Emmet, Teler, DIN 66131). The aggregates/agglomerates preferably have an equivalent spherical particle size (median particle size, MPS) in the range about 0.1-5.0 µm, e.g. about 0.2-4 µm, typically about 0.5-3.0 µm, as determined e.g. by sedimentation on a Sedigraph 5100 from Micromeritics. The aggregates'/agglomerates' BET surface area will typically be up to about 100 m^2/g, more typically up to about 80 m^2/g, e.g. up to about 50 m^2/g, e.g. up to about 30 m^2/g and typically at least about 15 m^2/g, e.g. at least about 20 m^2/g. The aggregates/agglomerates consist of a greater or smaller number of single crystals having an equivalent spherical particle size of, typically, about 0.01-0.50 µm.

It will be apparent to the skilled person that colloidal PCC can also occur as aggregates with a surface area of less than 10 m^2/g, but as mentioned above the expression "colloidal PCC" in the context of the present application is to be understood as PCC with the stated surface area of at least 10 m^2/g. Correspondingly, according to the present invention a PCC mixture in which a part of the mixture is colloidal PCC with a surface area of at least 10 m^2/g and a part of the mixture is "non-colloidal PCC" can be used, "non-colloidal PCC" being defined as PCC with a surface area of less than 10 m^2/g.

An example of a colloidal PCC product according to the invention is given in the table below:

Parameter	Value
Median particle size, MPS (µm)	1.5
Brightness (R_{457}-ISO, %)	95.8
Surface area (BET, m^2/g)	25.0

The particle size distribution of this PCC product is shown in Fig. 1, whilst Fig. 2 shows a SEM picture of typical aggregates.

The colloidal PCC can, if desired, be used alone, i.e. as sole filler or pigment, in the manufacture of paper, but will presumably normally be used with at least one further

filler or pigment. These further fillers and pigments can be selected among both non-colloidal PCC and other types of fillers. There is a wide variety of types of PCC with different crystal forms which are suited as a filler, e.g. scalenohedral PCC, rhombohedral PCC, needle-shaped PCC (aragonite) and spherical PCC (vaterite). Among other types of fillers and pigments which are suited for incorporation in paper, the following can be named: kaolin, calcined kaolin, talc, gypsum, ground marble, aluminium silicate, calcium silicate, magnesium silicate and other silicate-containing minerals, calcium sulphate, barium sulphate, titanium dioxide, zinc oxide, zinc carbonate, calcium sulfoaluminates (satin white), aluminium hydroxide, diatomaceous earth, plastic particles and organic pigments. Paper manufactured according to the present invention can, in addition to the colloidal PCC, suitably contain one or more such PCC or non-PCC fillers or pigments to obtain the desired paper properties. Preferred further fillers are non-colloidal PCC, kaolin, calcined kaolin, talc, gypsum, chalk, ground marble, silicate-containing minerals and calcium sulfoaluminates. Non-colloidal PCC, kaolin, calcined kaolin, chalk and ground marble are particularly preferred.

The finding which forms the basis of the invention, namely the fact that the porosity of paper can be regulated accurately by means of colloidal PCC, provides the advantage, however, that the relative amount of the colloidal PCC relative to other fillers and/or pigments, as well as the colloidal PCC's properties (especially the surface area), can be adjusted in each individual case in order to achieve the properties which are desired for the paper in question. It is thus clear that the amount of colloidal PCC which is to be used depends on the type of paper to be manufactured and on the type and amount of any other fillers. The amount of colloidal PCC to be used can therefore vary widely, i.e. from about 1% by weight of the total filler up to 100% of the total filler. The colloidal PCC will normally be used in an amount of at least 10% by weight, more typically at least 20% by weight, e.g. at least about 50% by weight, based on the weight of the total filler. The precise amount of colloidal PCC to be used in order to achieve the desired properties for a given paper, including a particular porosity, will be easily determined by the skilled person, e.g. by simply preparing a series of paper samples in which there are used different amounts of the colloidal PCC relative to the other fillers.

Typically, the amount of colloidal PCC used according to the invention will be at least about 1% by weight based on the total weight of the paper, more typically at least about 2% by weight, e.g. at least about 3% by weight, such as at least about 4% or 5% by weight. Depending on the total amount of filler in the paper and the proportion of the filler that is comprised by the colloidal PCC, the colloidal PCC can of course be present in significantly higher amounts, however.

According to the invention, the colloidal PCC can be used as a filler to regulate the porosity and printing properties of any type of paper, including e.g. wood-containing paper such as super-calendered (SC) paper/newsprint and wood-free paper such as fine paper. The invention is particularly suited for regulating the porosity and printing properties of uncoated paper, more particularly uncoated wood-containing paper, since these properties can be difficult to regulate in such paper compared to coated paper, where the porosity is controlled by the coating layer. In a preferred embodiment, the invention relates to the use of the colloidal PCC in the preparation of SC paper.

It will be known to persons skilled in the art of paper manufacturing that the terms "wood-containing" and "wood-free" refer to whether or not the lignin component of the ligno-cellulose wood fibres has been removed. These terms are used herein in accordance with their conventional meanings in the art, i.e. "wood-free" refers to cellulose fibres in which substantially all or at least most of the lignin has been removed, whereas "wood-containing" refers to ligno-cellulose fibres in which the lignin component has not been removed. While the specific amount of lignin that can be present in "wood-free" pulp may vary from country to country, this amount is relatively small. For example, in Finland wood-free paper is defined as paper in which less than 10% by weight of the pulp is groundwood or other lignin-containing pulp. In the present context, "wood-containing paper" thus refers to paper in which the fibres comprise a substantial lignin component, wherein typically at least about 5% by weight of the pulp is lignin-containing pulp, more typically at least about 10% by weight, such as at least about 15 or 20% by weight.

Removal of lignin to result in wood-free fibres can performed by means of various well-known processes, e.g. using the Kraft process or by sulphite pulping. Such

processes that remove lignin from the wood fibres result in higher quality, but also more expensive fibres.

In the case of SC paper, in particular SC-A paper, containing colloidal PCC according to the invention, the porosity can e.g. be reduced to a value of at most about 0.30 µm/Pas, e.g. at most about 0.28 µm/Pas, e.g. at most about 0.26 µM/Pas, e.g. at most about 0.24 µM/Pas, e.g. at most about 0.22 µM/Pas. In other words, the porosity of the paper can be reduced to a value around, or possibly even lower than, the value of the porosity of an equivalent paper prepared on the basis of kaolin; this is illustrated in Example 1.

The present invention also allows improved porosity values in SC-B paper. Thus, SC-B paper containing colloidal PCC according to the invention may have a porosity of at most about 0.60 µm/Pas, e.g. at most about 0.50 µm/Pas, e.g. at most about 0.40 µm/Pas, e.g. at most about 0.35 µm/Pas.

It will be known to persons skilled in the art that SC paper may be classified into one of several subcategories based on properties of brightness, filler level, roughness, sheet gloss and porosity. The top grade of SC paper is thus SC-A+. SC-A paper typically differs from SC-A+ in having a somewhat lower brightness, while SC-B typically differs from SC-A in having one or more of a lower brightness, a lower filler level, a lower sheet gloss and a higher porosity.

In the context of the present specification and claims, the SC paper grades SC-A, SC-A+ and SC-B are defined as follows.

<u>SC-A</u>

Brightness ≥ 64%

Filler level ≥ 30%

Roughness (0.5 bar) ≤ 2.0 µm

Roughness (1 bar) ≤ 1.5 µm

Porosity ≤ 0.3 µm/Pas

SC-A +

As SC-A above, but brightness ≥ 70%

SC-B

SC papers that do not fulfil the requirements for SC-A, but which fulfil the following requirements:

Brightness ≥ 60%

Filler level ≥ 15%

Roughness (0.5 bar) ≤ 3.0 μm

Roughness (1 bar) ≤ 2.5 μm

Porosity ≤ 0.6 μm/Pas

In the case of newsprint, the use of colloidal PCC according to the invention will make it possible to reduce the porosity of the paper to a value of at most about 20 µM/Pas, e.g. at most about 18 µM/Pas, e.g. at most about 16 µM/Pas; this is illustrated in Example 2. For SC paper, newsprint and other types of paper the porosity achieved in each case will depend among other things on the pulp used and on the amount and properties of the colloidal PCC and any other fillers used. The above mentioned porosity values for SC paper and newsprint, respectively, are therefore only to be taken as examples, the important feature of the invention being the possibility of regulating (reducing) the porosity relative to the porosity which would otherwise be achievable in a given paper using a filler according to the prior art.

Colloidal PCC can be prepared in a known manner by carbonating milk of lime (calcium hydroxide slurry) under suitable conditions. The following conditions are to be regarded as a non-limiting example of the preparation of colloidal PCC:

Burnt lime having a reactivity (DIN T_{60}) of between 10 sec. and 5 min. is slaked in 40°C warm water using a water/lime ratio of 4:1. The thus-prepared milk of lime is diluted to 40% dry matter content, after which it is screened through a 500 µm screen.

After screening, the milk of lime is cooled to 20°C and carbonated in an appropriate gas flow reactor using flue gas or a CO_2-air mixture typically containing 20% CO_2. Carbonation is continued until the pH has fallen below 8.

At a gas flow of 2.5 m^3 flue gas per m^3 reactor volume the reaction will occur over a period of about 3 hours. After carbonation is completed the colloidal PCC is screened through a 45 µm screen.

The invention is further illustrated by the following non-limiting examples.

In the examples below, the following standards were used for determining paper properties:

Gram weight: Scan-P 6:75
Thickness: Scan-P 7:96
Density: Scan-P 7:96
Gloss: Tappi T480 om-92
Brightness: ISO 2470
Opacity: ISO 2471
Roughness: Scan-P 76:95
Porosity: PPS method

All amounts are by weight unless otherwise indicated.

EXAMPLE 1: REGULATION OF POROSITY IN SC PAPER

The following pigments were tested in SC paper:

	Kaolin reference Filler - M (ECC International)	Rhombohedral PCC Standard product (Faxe Paper Pigments A/S)	Colloidal PCC Experimental product (Faxe Paper Pigments A/S)
Brightness (R_{457}-ISO, %)	78.9	97.0	95.9
MPS (µm)	3.3	1.8	1.1
BET (m^2/g)	9.0	6.2	25

The test was carried out on a pilot paper machine with filler levels of 27, 30 and 33%.

The fibers were of Scandinavian origin and consisted of:

TMP (thermomechanical pulp) and GW (groundwood)	85%
Kraft (cellulose fibers processed by the "kraft" process)	15%

The following chemicals were used in the manufacturing process:

Retention agent	none
Other	none

pH adjusted to 7.3 by addition of H_3PO_4.

For comparison purposes the results for paper are interpolated to 30% filler after calendering. The results are shown in the table below.

	Kaolin reference	Rhombohedral PCC	Colloidal PCC
Gram weight (g/m^2)	55	56	56
Thickness (µm)	49	54	55
Density (g/m^2)	1.123	1.030	1.020
Gloss (75°, %)	35	32	36
Brightness (R_{457}-ISO, %)	69.6	76.3	72.5
Opacity (%)	86.8	90.0	85.9
Roughness (µm)	1.48	1.48	1.46
Porosity µm/Pas	0.19	0.32	0.21

It can be seen from the above table that colloidal PCC surprisingly is capable of lowering the porosity of the paper from 0.32 µm/Pas using a standard PCC to 0.21 µm/Pas with colloidal PCC, which is on a par with the kaolin reference.

EXAMPLE 2: REDUCTION OF POROSITY OF NEWSPRINT USING COLLOIDAL PCC AS FILLER

The following pigments were tested in newsprint:

	Reference Calcined Kaolin (Ansilex from Engelhard)	Faxe Chalk 89 Chalk (Faxe Kridt A/S)	Rhombohedral PCC (Faxe Paper Pigments A/S)	Colloidal PCC Experimental product (Faxe Paper Pigments A/S)
Brightness (R_{457}-ISO, %)	89.6	87.4	96.2	95.7
MPS (µm)	0.9	1.5	1.2	1.1
BET (m^2/g)	15.0	3.2	9.2	23.0

The test was carried out on a pilot paper machine with filler levels from 2-10%.

The fibres consisted of:

Unbleached TMP (thermomechanical pulp)	95%
Bleached cellulose prepared by the sulphate process	5%

The following chemicals were used in the preparation:

Retention agent	Percol 230L (cationic polyacrylamide from Allied Colloids)
Other	none

pH adjusted to 7.3 by addition af H_2SO_4.

For comparison purposes the results for paper are interpolated to 4% filler. The results are given in the following table, the gram weight of the papers being 46 g/m^2.

	Reference Calcined Kaolin (Ansilex from Engelhard)	Faxe Chalk 89 Chalk (Faxe Kridt A/S)	Rhombohedral PCC (Faxe Paper Pigments A/S)	Colloidal PCC Experimental product (Faxe Paper PigmentsA/S)
Thickness (µm)	106	106	105	105
Roughness (µm)	5.2	6.2	6.2	6.2
Porosity (µm/Pas)	17	21	20	15
Brightness (R_{457}-ISO, %)	63.5	61.1	61.6	60.5
Opacity (%)	90.2	89.4	89.8	90.6

It can be seen from the table above that colloidal PCC surprisingly is able to lower the porosity of the paper from 21 µm/Pas with a standard PCC to 15 µm/Pas with colloidal PCC, which is lower than the kaolin reference at 4% filler level.

Conclusion

By using colloidal PCC as filler the porosity of the paper is lowered significantly. The amount of colloidal PCC in the paper can thereby be varied as required, so that the porosity and thereby also the printing properties can be regulated precisely. The colloidal PCC can thus be used as required instead of or in combination with other conventional fillers and pigments in order to achieve the desired porosity.

EXAMPLE 3

A pigment mixture consisting of 50 parts (by weight) fine scalenohedral PCC, 30 parts fine rhombohedral PCC and 20 parts colloidal PCC was tested in production scale as a filler in SC-A grade paper at a commercial paper mill. The PCC pigment mixture was pH-stabilised by addition of a small amount of phosphoric acid in order to avoid the need for acid addition on the paper machine for pH-control. The properties of the PCC mixture and the reference clay filler used in the trial are listed in the table below.

	Reference kaolin clay (European filler grade)	Experimental PCC mixture (Faxe Paper Pigments A/S)
Brightness (R_{457}-ISO %)	79.2	94.1
MPS (µm)	1.38	1.62
BET surface area (m^2/g)	11.7	10.8

The pulp furnish composition was 50 parts deinked pulp (DIP), 40-45 parts groundwood (GW) and 5-10 parts Kraft pulp.

The trial PCC mixture was tested at a constant total filler level with two levels of PCC addition. The balance to give the total amount of filler is reference clay and filler introduced with the DIP (recycled paper).

The properties of the papers resulting from the trial are listed in the table below.

	Reference	Trial 1	Trial 2
Added PCC[1]	0 %	10 %	20 %
Added clay[1]	32 %	22 %	12 %
Analysed $CaCO_3$ content[1]	1.5 %	13.4 %	24.2 %
Analysed clay content[1]	37.6 %	25.6 %	15.2 %
Gram weight	57 g/m^2	56 g/m^2	56 g/m^2
Roughness TS (0.5 bar)	1.70 µm	1.75 µm	1.65 µm
Roughness WS (0.5 bar)	1.70 µm	1.70 µm	1.55 µm
Porosity (PPS)[2]	0.122 µm/Pa·s	0.197 µm/Pa·s	0.228 µm/Pa·s
Gloss 75°, TS MD	50 %	45 %	45 %
Gloss 75°, WS MD	48 %	49 %	49 %
Brightness R_{457}-ISO	66.4 %	70.1 %	72.1 %
Opacity	92.1 %	92.5 %	91.8 %

1) By weight, based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction

2) PPS = Parker-Print-Surf method

The runnability of the paper machine remained good during the two-day trial period and it was possible to increase the production capacity by 1.5%. The Hydrocol™ two-component retention system was used on the paper machine. The amount of cationic polymer could be reduced during the trial as the PCC pigment mixture was easier to retain than the reference clay. The pH in the paper machine headbox was 7.4 prior to the trial and it increased only slightly (to 7.6) during the trial.

The paper produced during the trial showed excellent results in full-scale commercial printing. It is remarkable that the paper brightness has been increased by 6 percentage points without any loss in opacity. The resulting 72% brightness is close to the superior SC-A+ quality.

EXAMPLE 4

A pigment mixture consisting of 80 parts (by weight) fine rhombohedral PCC and 20 parts colloidal PCC was tested in production scale as a filler in SC-B grade paper at a commercial paper mill. The rhombohedral PCC and the colloid PCC had BET surface areas of approximately 7 and 20 m^2/g, respectively, to provide a mixture having an overall BET surface area of 9.1 m^2/g as indicated below. The PCC pigment mixture

was pH-stabilised by addition of a small amount of phosphoric acid in order to avoid the need for acid addition on the paper machine for pH-control. The properties of the PCC mixture and the reference fillers used in the trial are listed in the table below.

Filler:	Reference kaolin clay (European filler grade)	Reference PCC	Experimental PCC mixture (Faxe Paper Pigments A/S)
Brightness (R_{457}-ISO %)	76.6	96.2	95.4
MPS (μm)	2.13	1.70	1.31
BET (m^2/g)	11.9	8.6	9.1

The pulp furnish composition was 30-35 parts deinked pulp (DIP), 10-15 parts chemothermomechanical pulp (CTMP) and groundwood (GW), adding up to a total of 100 parts.

The trial PCC mixture was tested at a constant total filler level with two levels of PCC addition. The balance to give the total amount of filler is reference clay and filler introduced with the DIP (recycled paper).

The properties of the papers resulting from the trial are listed in the table below.

	Reference	Trial 1	Trial 2
Added Faxe PCC[1]	0 %	11 %	18 %
Added reference PCC[1]	11 %	0 %	0 %
Added clay[1]	11 %	11 %	4 %
Analysed $CaCO_3$ content[1]	15.8 %	14.8 %	22.4 %
Analysed clay content[1]	17.8 %	17.4 %	11.2 %
Gram weight	57 g/m^2	56 g/m^2	56 g/m^2
Roughness TS (0.5 bar)	2.80 μm	2.90 μm	2.90 μm
Roughness WS (0.5 bar)	2.60 μm	2.80 μm	2.80 μm
Porosity (PPS)	0.570 μm/Pa·s	0.514 μm/Pa·s	0.554 μm/Pa·s
Gloss 75°, TS MD	27 %	27 %	26 %
Gloss 75°, WS MD	24 %	25 %	24 %
Brightness R_{457}-ISO	63.4 %	62.6 %	64.8 %
Opacity	96.4 %	95.9 %	96.0 %

1) By weight, based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction.

The runnability of the paper machine remained good during the two-day trial period and it was possible to increase the production capacity by 1.3%. The Hydrocol™ two-component retention system was used on the paper machine. The amount of cationic polymer could be reduced during the trial, as the PCC pigment mixture was easier to retain than the reference clay. The amount of blue and yellow colour could be reduced as well. The pH in the paper machine headbox was 7.3 prior to the trial and it was stable at 7.2±0.1 during the trial.

The paper produced during the trial showed excellent results in full-scale commercial printing. The pulp bleaching was reduced in order to keep the paper brightness within the production specifications. The reduced amount of bleaching chemicals is an advantageous cost saving for the paper mill and environmentally beneficial.

EXAMPLE 5

A pigment mixture consisting of 80 parts (by weight) fine rhombohedral PCC and 20 parts colloidal PCC was tested in production scale as a filler in SC-A grade paper at a commercial paper mill. The PCC pigment mixture was pH-stabilised by addition of a small amount of phosphoric acid in order to avoid the need for acid addition on the paper machine for pH-control. The properties of the PCC mixture and the reference clay fillers used in the trial are listed in the table below. The paper mill alternates between use of two clays in their normal production.

Filler:	Reference kaolin clay (European filler grade)	Experimental PCC mixture (Faxe Paper Pigments A/S)
Brightness (R_{457}-ISO %)	80.7	94.1
MPS (μm)	1.79	1.62
BET (m^2/g)	15.4	10.8

The pulp furnish composition was 75 parts deinked pulp (DIP), 20 parts groundwood (GW) and 5 parts Kraft pulp.

The trial PCC mixture was tested at a constant total filler level with all fresh filler added being PCC. The balance to give the total amount of filler is filler introduced with the DIP (recycled paper). Paper was made in three gram weights: 48, 52 and 56 g/m^2. For the sake of simplicity only results for 56 g/m^2 are shown. The results at the other gram weights were similar.

The properties of the papers resulting from the trial are listed in the table below.

	Reference	Trial
Added PCC[1]	0 %	17 %
Analysed $CaCO_3$ content[1]	3.5 %	18.3 %
Analysed clay content[1]	32.9 %	17.4 %
Gram weight	57 g/m^2	57 g/m^2
Roughness TS (0.5 bar)	2.4 µm	2.20 µm
Roughness WS (0.5 bar)	2.65 µm	2.55 µm
Porosity (PPS)	0.252 µm/Pa·s	0.367 µm/Pa·s
Gloss 75°, TS MD	32.9 %	31.7 %
Gloss 75°, WS MD	26.1 %	29.2 %
Brightness R_{457}-ISO	66.0 %	66.4 %
Opacity	94.1 %	95.6 %

1) By weight based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction.

The runnability of the paper machine remained good during the two-day trial period and it was possible to increase the production capacity by 1.2%. The Hydrocol™ two component retention system was used on the paper machine. The amount of cationic polymer could be reduced by approx. 20% during the trial as the PCC pigment mixture was easier retained than the reference clay. The pH in the paper machine headbox was 7.6 prior to the trial and it increased only slightly (to 7.7) during the trial.

The paper produced during the trial showed excellent results in full-scale commercial printing. It is remarkable that the paper mill had to totally stop bleaching their DIP in order to keep the brightness within the production specifications. This is a big economic advantage and also environmentally beneficial.

EXAMPLE 6

A number of fillers and filler mixtures were tested in a dynamic sheet former trial.

The fillers were three PCCs from Faxe Paper Pigments A/S, Denmark (a fine rhombohedral PCC, a fine scalenohedral PCC, and a colloidal PCC), and a kaolin clay from Dorfner. The properties of the fillers used in the trial are listed in the table below.

Filler	Brightness (R_{457}-ISO %)	MPS (μm)	BET (m^2/g)
Fine rhombohedral PCC	94.6	0.90	7.9
Fine scalenohedral PCC	95.7	2.13	9.3
Colloidal PCC	95.4	1.40	28.1
Kaolin clay (Dorfner)	81.7	2.02	8.4

Handsheets were made on a dynamic sheet former from Fibertech AB. The pulp furnish consisted of 50 parts groundwood, 30 parts DIP and 20 parts Kraft pulp. The target filler level was 35% by weight of the total weight of the paper. The results are listed below. The target gram weight of the handsheets was 56 g/m^2 (The actual gram weights varied between 53.2 and 58.1 g/m^2). Handsheets were made at three target filler levels, which were 30%, 33% and 36% filler by weight based on the total weight of the paper. The paper quality parameters were interpolated to a 35% filler level and the results are listed below.

Trial No:	1	2	3	4	5	6	7
Fine rhombohedral PCC[1]	100	80	70	50			
Fine scalenohedral PCC[1]					100	80	50
Colloidal PCC[1]		20	30	50		20	
Kaolin Clay[1]							50
Analysed $CaCO_3$ content[2] (%)	32.5	32.9	32.3	32.7	32.9	32.8	18.8
Analysed clay content[2] (%)	2.5	2.1	2.7	2.3	2.1	2.2	16.2
Gram weight (g/m^2)	54.2	53.9	55.0	56.1	57.0	56.5	56.9
Roughness TS (1 bar) (μm)	1.33	1.35	1.31	1.35	1.28	1.28	1.16
Porosity (PPS) (μm/Pa·s)	0.276	0.271	0.265	0.252	0.337	0.259	0.236
Gloss 75°, TS MD (%)	32.0	35.1	37.2	40.1	39.7	40.2	46.7
Brightness R_{457}-ISO (%)	70.2	69.5	68.6	67.6	68.8	67.4	66.6
Opacity (%)	92.7	93.0	93.8	92.9	93.5	92.9	92.7

1) parts by weight, 2) By weight based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction.

CLAIMS

1. A process for regulating the porosity and printing properties of uncoated paper, the process comprising using a sufficient quantity of colloidal PCC having a BET surface area of 10-100 m^2/g as a filler to achieve a desired porosity of the paper.

2. A process according to claim 1, wherein the paper is wood-containing paper.

3. A process according to claim 2, wherein the paper is SC paper, in particular SC-A paper, and wherein colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.30 µm/Pas, e.g. at most 0.28 µm/Pas, e.g. at most 0.26 µm/Pas, e.g. at most 0.24 µm/Pas, e.g. at most 0.22 µm/Pas.

4. A process according to claim 2, wherein the paper is SC-B paper, and wherein colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.60 µm/Pas, e.g. at most 0.50 µm/Pas, e.g. at most 0.40 µm/Pas, e.g. at most 0.35 µm/Pas.

5. A process according to claim 2, wherein the paper is newsprint, and wherein colloidal PCC is used in an amount sufficient to achieve a porosity of at most 20 µm/Pas, e.g. at most 18 µm/Pas, e.g. at most 16 µm/Pas.

6. A process according to any of the preceding claims, wherein the colloidal PCC has a BET surface area of 15-50 m^2/g.

7. A process according to claim 6, wherein the colloidal PCC has a BET surface area of 20-30 m^2/g.

8. A process according to any of the preceding claims, wherein colloidal PCC is incorporated into the paper in an amount of at least about 1% by weight based on the total weight of the paper.

9. A process according to claim 8, wherein colloidal PCC is incorporated into the paper in an amount of at least about 2% by weight based on the total weight of the paper.

10. Paper containing colloidal PCC having a BET surface area of 10-100 m^2/g as a filler.

11. Paper according to claim 10, comprising at least one further filler selected from non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

12. Paper according to claim 10 or 11, wherein the colloidal PCC has a BET surface area of 15-50 m^2/g, e.g. 20-30 m^2/g.

13. Paper according to any of claims 10-12, said paper being uncoated.

14. Paper according to any of claims 10-13, wherein the colloid PCC is present in an amount of at least about 1% by weight, e.g. at least about 2% by weight, based on the total weight of the paper.

15. Uncoated wood-containing paper containing colloidal PCC.

16. SC paper containing colloidal PCC and having a porosity of at most 0.30 µm/Pas, e.g. at most 0.28 µm/Pas, e.g. at most 0.26 µm/Pas, e.g. at most 0.24 µm/Pas, e.g. at most 0.22 µm/Pas.

17. SC paper according to claim 16, wherein the paper is SC-A paper.

18. SC-B paper containing colloidal PCC and having a porosity of at most 0.60 µm/Pas, e.g. at most 0.50 µm/Pas, e.g. at most 0.40 µm/Pas, e.g. at most 0.35 µm/Pas.

19. Newsprint containing colloidal PCC and having a porosity of at most 20 µm/Pas, e.g. at most 18 µm/Pas, e.g. at most 16 µm/Pas.

20. Paper according to any of claims 15-19, comprising at least one further filler selected from non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

21. Paper according to any of claims 15-20, wherein the colloidal PCC has a BET surface area of 10-100 m^2/g, e.g. 15-50 m^2/g, e.g. 20-30 m^2/g.

22. A pigment mixture suitable for paper manufacture and comprising colloidal PCC having a BET surface area of 10-100 m^2/g in combination with at least one filler selected from the following pigments: kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

23. A pigment mixture suitable for paper manufacture and comprising a combination of colloidal PCC having a BET surface area of 10-100 m^2/g and non-colloidal PCC.

24. A pigment mixture according to claim 22 or 23, wherein the colloidal PCC has a BET surface area of 15-50 m^2/g, e.g. 20-30 m^2/g.

25. A pigment mixture according to any of claims 22-24, wherein the colloidal PCC comprises aggregates/agglomerates having an equivalent spherical particle size in the range 0.1-5.0 μm, e.g. 0.2-4 μm, typically 0.5-3.0 μm, wherein the aggregates/agglomerates consist of single crystals having an equivalent spherical particle size of about 0.01-0.50 μm.

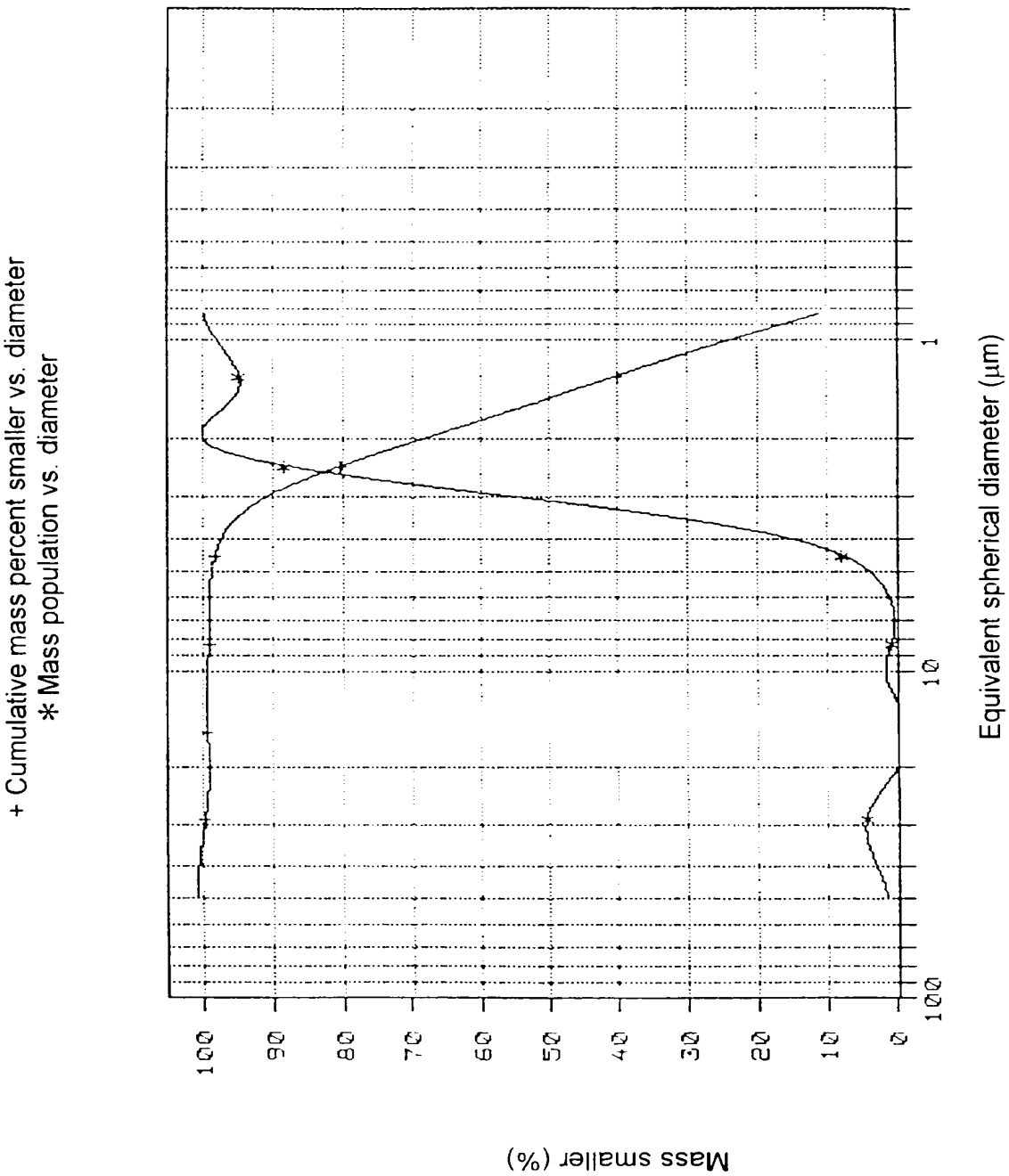
+ Cumulative mass percent smaller vs. diameter
* Mass population vs. diameter
Mass smaller (%)
100
90
80
70
60
50
40
30
20
10
0
Equivalent spherical diameter (µm)
1
10
100

Fig. 1

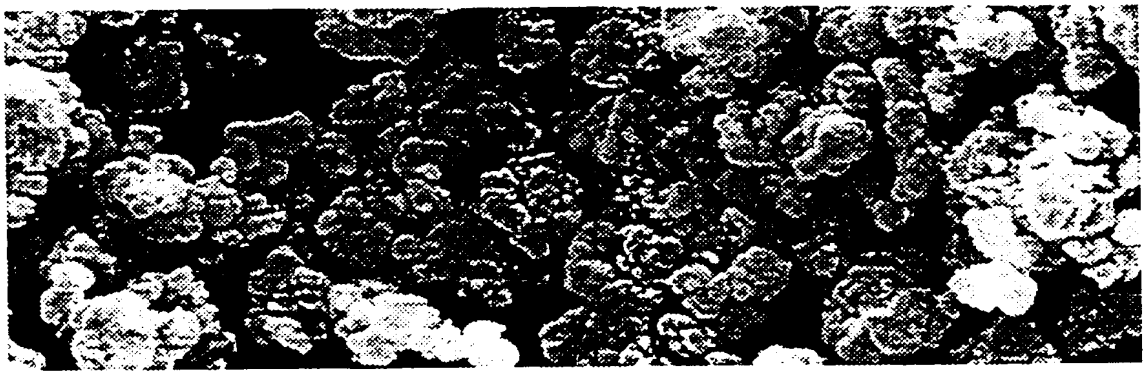

Fig. 2